CHAPTER III
EXPERIMENTAL

3.1 Materials and Equipment

Raw Material:

Nata de coco (coconut gel)

Equipment:

1. Gas separation unit
2. Freeze dryer (Lyolab LT3S, Lyophilization system, Inc. USA)
3. Thermogravimetric/Differential Thermal Analyzer (Perkin Elmer Pyris Diamon TG/DTA)
4. Differential Scanning Calorimeter (DSC 822, Metler Toledo)
5. X-ray diffractometer (XRD, Rigaku, SmartLab)
6. Gas pycnometer (Ultrapycnometer 1000)
7. Scanning Electron Microscope (SEM, JEOL, JSM-5410LV), (EDX, Oxford ISIS series300)
8. Fourier transform infrared spectrometer (Spectrum one FTIR, Perkin Elmer, Universal ATR Sampling Accessory)
9. Analytical balance
10. Oven
11. Desiccator
12. Blender

Software:

1. Essential FTIR

Chemicals:

1. Sodium hydroxide (NaOH, LABSCAN ASIA CO.)
2. Silver nitrate ($AgNO_3$, VR BIOSCIENCE CO.)

3.2 Experimental Procedures

3.2.1 Measurements

3.2.2.1 Thermogravimetric/Differential Thermal Analyzer (TG/DTA)

Thermal gravimetric analysis were performed using Perkin Elmer Pyris Diamon TG/DTA instrument in a nitrogen atmosphere (flow rate 10 ml/min). The sample was heated from 50°C to 110°C, then were hold at 110°C for 30 min and heated from 110°C to 450°C at a heating rate of 10°C/min.

3.2.2.2 Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetric experiments were carried out by using a Differential Scanning Calorimeter (DSC 822, Metler Toledo) in a nitrogen atmosphere (flow rate 20 ml/min). A 4-8 mg of sample was placed in a tightly sealed aluminum pan. The sample was subjected to run against an empty pan as a reference at a heating rate of 10°C/min in the temperature range 0°C –300 °C.

3.2.2.3 Fourier Transform Infrared Spectroscopy (FTIR)

Infrared spectra of bacterial cellulose membranes were characterized by the Spectrum one FTIR (Perkin Elmer). Each sample was analyzed to determine the structural characteristics in the range of 515-4000 cm^{-1} at room temperature by using a Universal ATR Sampling Accessory. Moreover, the sample impregnated with silver ions was further investigated the reaction with CO_2 by soaking the sample into the CO_2 contained bottle overnight and then this sample was analyzed by FTIR (Universal ATR Sampling Accessory) technique.

3.2.2.4 Scanning Electron Microscopy (SEM-EDX)

The morphologies of the samples were studied using Scanning Electron Microscope (SEM, JEOL, JSM-5410LV). Prior to analysis, the samples were cracked in liquid nitrogen into the small pieces and sputtered coated with a thin layer of gold under vacuum, then examined at 35x and 100x magnifications for every samples and at 500x and 3,500x magnifications for the samples impregnated with silver ions, of which the last magnification was used for the sample without silver ions as well in order to improve that these membranes do not show the presence of silver nitrate in bacterial cellulose' fibers. Additionally, the chemical compositions on the

surface of membranes can be proved by the EDX mode (EDX, Oxford ISIS series300). Two regions of surface area of the membranes were randomly chosen for analyzing.

3.2.2.5 X-ray Diffraction (XRD) Analysis

The crystalline structure of bacterial cellulose membrane was investigated by the XRD pattern which was recorded by X-ray diffractometer (XRD, Rigaku) equipped with filter Cu K_β radiation at a scanning rate of 8°/min ranging from 10° to 80° (2Θ angle).

3.2.2.6 Gas Pycnometer (Ultrapycnometer 1000)

Gas pycnometer (Ultrapycnometer 1000) was used for determination of the average true density of the membranes at each ratio of dried Nata de coco to water.

3.2.2 Gas Separation Study

All of bacterial cellulose membranes with and without silver ions were tested in a single gas (carbon dioxide or methane) measurement. The experiments were conducted for two hours at room temperature and the pressure difference between the feed and the permeating sides (ΔP) was maintained at 20 psi. The gas separation unit for this study is schematically shown in figure 3.1.

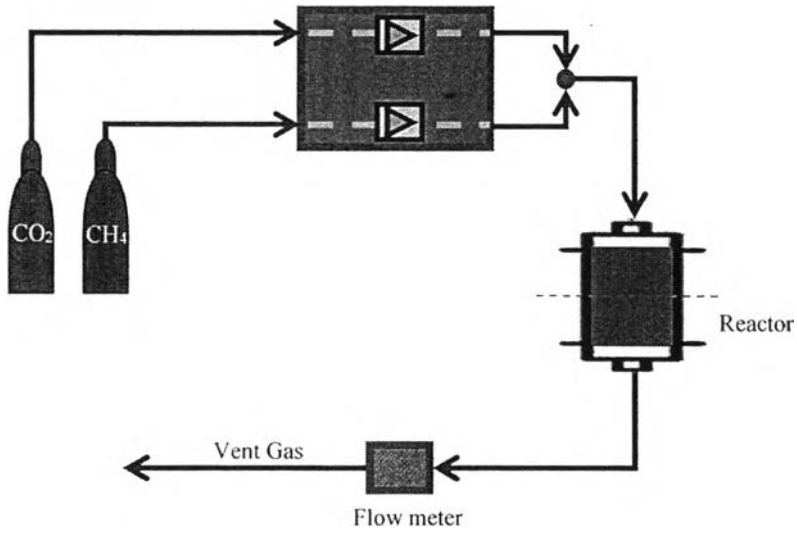

Figure 3.1 The unit of separation study.

3.2.3 Experimental

3.2.3.1 Purification of Coconut Gel (Nata De Coco)

Nata de coco was cut into the small pieces and soaked in boiled water for fifteen minutes with continuous stirring, followed by soaking and continuous stirring in 0.1 M NaOH at 80°C until its color was clear. The alkaline solution was changed every two hours. After that Nata de coco was washed several times with distilled water until the pH of water became neutral (Chawla *et al.*, 2009). This purified gel was stored in distilled water prior to use.

3.2.3.2 Preparation of Bacterial Cellulose Membranes

The purified Nata de coco was blended by the blender and separated water out. Afterward, it was dried in an oven for 6 hours and then blended it again to obtain bacterial cellulose powder. The various amounts of water, which are 7, 10, 13, 15, 17 and 20 ml, were added into 1 g of the bacterial cellulose powder for each condition and the samples were stirred until the powder was swollen enough to be continuous matrix membranes. The samples were then freeze-dried by a freeze-dryer to finally obtain bacterial cellulose membranes.

3.2.3.3 Preparation of Bacterial Cellulose Membranes with Silver Ions

Certain amount of water, which is 9 ml, was added into 1 g of the bacterial cellulose powder and stirred until the powder was swollen enough to be continuous matrix membranes. Various concentrations of silver nitrate solution (1 ml), (0.1M, 0.5M and 1.0M) were added into the swollen samples. After that the samples were then freeze-dried by the freeze-dryer to obtain bacterial cellulose membranes with silver ions.

3.2.4 Characterizations

Thermal properties of bacterial cellulose membranes were observed by Thermogravimetric Analyzer (TGA) and Differential Scanning Calorimeter (DSC). The chemical structure of bacterial cellulose was revealed by FTIR. The morphology of the membranes was characterized using a scanning electron microscopy (SEM). The surface compositions of the membranes were determined using the EDX mode. The average true density of bacterial cellulose membrane was determined by gas

pycnometer. The crystalline structure of bacterial cellulose structure was studied by X-Ray Diffraction (XRD) analysis.

3.2.5 CO_2/CH_4 Separation Study

All of bacterial cellulose membranes with and without silver ions were tested in a single gas (carbon dioxide or methane) measurement. Carbon dioxide (CO_2) and methane (CH_4) gas were of high purity and used as received. The experiments were performed for two hours at room temperature and the pressure difference between the feed and the permeating sides (ΔP) was maintained at 20 psi. The area of the membrane in contact with the gas was 0.5024 cm^2. The equilibrium state was obtained by measuring the constant permeate rate. When it reached the steady–state, individual gas flow rate was measured by a gas flow meter. The obtained data were used to calculate the gas permeance and selectivity (Nicharat *et al.*, M.S.Thesis).

The permeance of the permeated gas can be determined by the following equation:

$$(\frac{P}{\delta})_i = \frac{Q_i \, x \, 14.7x10^6}{(A)x(\Delta P)x76}$$

Where $(\frac{P}{\delta})_i$ = permeance of gas "i" (GPU),

P = permeability of gas 'i' (10^{-10} cm^3 (STP) cm/cm^2 s cm Hg)

(1 Barrer = 10^{-10} cm^3 (STP) cm/cm^2 s cm Hg = 7.5×10^{-18} m^2 s^{-1} Pa^{-1}),

δ = thickness of membrane (μm),

Q_i = volumetric flow rate of gas 'i' (cm^3/sec),

A = membrane area (cm^2), and

ΔP = pressure difference between the feed side and the permeating side (psi).

The ideal separation factor (Gas selectivity, $S_{A/B}$) for component A and B is defined according to the following equation (Nicharat et al., M.S.Thesis):

$$S_{A/B} = \frac{P_A}{P_B}$$

Where P_A = the permeance of component A, and

P_B = the permeance of component B.